Outline of the Research Work

(Synopsis)

Studies on Synthesis, Characterization and Pharmacological Evaluation of Multidentate Schiff's bases of di, trivalent Metal complexes

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Studies on Synthesis, Characterization and Pharmacological Evaluation of Multidentate Schiff's bases of di, trivalent Metal Complexes

Introduction:

Schiff bases are an unusual type of ligands having biological activities due to remarkable co-ordination modes towards transition metals and existence of an imine or azomethine (-C=N-) linkage.This type of compound was first synthesized by German scientist Hugo Schiff in 1864 and hence named after him as Schiff base [1-3].Schiff-bases starting from various amines have been investigated by different methods [4,5]and have been shown to have interesting applications in catalytic reactions, materialschemistry, and in industry. Schiff base obtained after condensation of Aliphatic and Aromatic aldehyde or ketone and the primary amine, are stated as flexible ligands for co-ordination chemistry. This important type of ligands contains in their structure as donor centers necessary for metal ions to project different geometrieswith other ligands. Schiff bases are mainly bidentate, tridentate, tetradentate or polydentate ligands capable of forming very stable complexes with their transition metals. They can only act as complexing ligands if they bear a functional group, usually the hydroxyl group, which is sufficiently near the site of condensation in such a way that a five or six membered ring can be formed when reacting with a metal ion.Schiff bases are called versatile ligands as they coordinate with various metal ions in several oxidation states and geometries. Schiff bases are found to produce complexes with most of the transition metals and with lanthanides.

Schiff bases originated from the aromatic amines and aromatic aldehydes have a wide variety of applications in many fields such as analytical, inorganic, biological [6,7].Transition metal complexes with Schiff bases have expanded tremendously and embraced wide and diversified subjects comprising vast areas of metal organic compounds and various aspects of bio co-ordination chemistry. The complexes of the Schiff-bases are of great consideration due to their stability, electron donating capacity, optical non linearity, catalytic, photochromic, and biological activity.These practical activities are all based on the co-ordination of Schiff-bases with the metalions. An interesting class of Schiff-base complexes is that obtained starting from aminoacids [8]. Amino acids are dynamically implicated in a part of biological processesand they possess the $–NH_2$ and –COOH coordinating sites, which can be bonded with aldehydes/ketones to synthesize Schiff-bases which are easily coordinated with the metalions.

In recent years much effort has been put in synthesis and characterization of mono and bi-nuclear transition metal complexes [9]. Schiff bases are used in optical and electrochemical sensors, as well as in various chromatographic methods to enable detection of enhanced selectivity and sensitivity [10-12]. Among the organic reagents actually used, Schiff bases possess several characteristics such as chemical properties, Structural similarities with natural biological substances, relatively simple preparation procedures and the synthetic flexibility that enables design of suitable structural properties [13]. Schiff bases are widely applicable in analytical determination, using reactions of condensation of primary amines and carbonyl compounds in which the azomethine bond is formed using complex forming reactions or utilizing the variation in their spectroscopic characteristics following the changes in pH and solvent [14]. Schiff bases play important role in co-ordination chemistry as they easily form

stable complexes with most transition metal ions [15]. In organic synthesis, Schiff base reactions are useful in making carbon-nitrogen bonds.

The Schiff bases and their metal complexes exhibit various biological activities like antibacterial, antifungal, antileukemic, anti-inflammatory, analgesic, antioxidant, anticancer, antitumor, acetylcholinesterase inhibition, urease inhibition etc. [16,17]. They also exhibit clinical, biochemical and pharmacological properties [18]. Schiff bases have tremendous importance as catalyst, dyes, pigments, agrochemicals, corrosion inhibitors, polymeric stabilizers etc. [19].

Thus Schiff bases occupy many important biological and pharmaceutical activities and they are distinguished for their pharmacological action and wide-range applications in the food industry.

Review of Literature:

1. Santosh Kumar and coworker synthesized Schiff base from 4-amino benzene sulphonamide and substituted aromatic aldehydes.[20]

2. Muhammad Aqeel Ashraf and co-worker, reported a series of Schiff bases from 2-amino-Benzthiazole, 4-amino-Salicylic acid and 4-aminophenol.[21]

3. Muhammad Aslam, *et al* [22] synthesized Schiff base by mixing of Aminophenol with 4-chloroacetophenon or 4-hydroxyacetophenone and the reaction mixture was refluxed for 3 h with stirring at 70^{o}C after adding 3-4drops of conc.H_2SO_4. 4-Chloro-2-oxo-2H-chromene-3-carbaldehyde was made to react with different anilines in rectified spirit to yield a series of Schiff bases ofthe type 4-chloro-3-(substituted-phenylimino)methyl)-2H-chromen-2-one reported by S. Bairagi*etal.*[23] Bag *etal* have synthesized a series of Schiff bases of benzidene with series of substituted aromatic aldehydes and examined the mercuration reaction.[24]

4. K. Mounika, B. Anupama and co-worker, prepared some Schiff basses by treating of 3-ethoxy Salicylaldehyde and 2-amino benzoic in ethanol. [25]

5. Vatsala Pawar, Sunil Joshi & V. Uma, synthesized macrocyclic ligands by taking equimolar ratios of Acetylacetone in ethanol with the solutions of Semicarbazide hydrochloride in hot water (dil.NaOH) and Thiosemicarbazide in ethanol respectively and then both were added drop wise in 25 ml of Ethanol underconstant stirring for at least 3 hours.[26]

6. Vivek Tiwari, Rashmi Singhai and A.P. Mishra, Synthesized some Schiff bases metal complexes of Ni(II) andCu(II) with Schiff bases '4- dimethylaminobenzylidene-4-chloroaniline'.[27]

7. B. K. Rai, synthesized a series of metal complexes with 2-methyl-3-phenyl quinazolin-4 (3H) one semicarbazone and its thiosemicarbazone analogue. [28]

8. N. Raman and co-workers [29], prepared a series of Schiff bases with 4- amino antipyrine and 3-hydroxy-4-nitrobenzaldehyde derivatives and its metal complexes with Cu(II), Ni(II),

Co(II), Mn(II), Zn(II), Cd(II), Hg(II) and V(IV) and studied its chemotherapeutic use as DNA cleavage and antimicrobial activity.[29]

9. S. Bawa and coworker synthesized A series of 4-substituted-emoni methyltetrazolo[1,5a] quinoline with appropriate aromatic amine by refluxing in dioxane. They have been evaluated for their anti-inflammatory and antimicrobial activities. [30]

10. Vijay Aanandhi*et al* have reported the synthesis of a series of 1-(5-substituted-2-oxoindolin-3-ylidene)-4-(substituted-pyridin-2yl)thiosemicarbazide derivatives. These compounds were screened for *in vitro* antibacterialand antifungal activity against *B.subtilis ,S. aureus, E.coli, P. aeruginosa, C. albicans,* and *A. niger.* All thecompounds were reported to exhibit moderate to good antibacterial and antifungal activity. [31]

11. Z.H. Chohan and S. Mushtaq synthesized A series of biologically active pyrazine derived Schiff base ligandshave been synthesized by the condensation reaction of 2-aminopyrazine with salicylaldehyde and acetamido benzylaldehyde. Then their Co(II), Ni(II) & Zn (II) complexes have been prepared. The biological evaluation of the simple uncomplexed ligand in comparison to their complexes have been determined against bacterial strains namely *Escherichia coli, Staphylococcus aureous* and *Pseudomonas aeruginosa*. [32]

12. Natarajan Raman *et al* [33] have reported the synthesis of a novel 14-membered macrocyclic Schiff base derivedfrom 3-cinnamalideneacetanalide and *o*-phenylenediamine which acts as a tetradentate and strongly conjugatedligand to form a cationic solid complex with Cu(II)/Ni(II)/Co(II) and /Zn(II). The ligand and the complexes were characterized by the usual spectral and analytical techniques. The antimicrobial tests were also recorded and gave good results in the presence of metal ions in the ligand system.

13. May Lee Low [34] has studied the synthesis of two new Schiff bases followed by complexation (with Cu) , electrochemistry, antibacterial activity and cytotoxic assay. The Schiff bases and their complexes have been characterized by using several physicochemical and spectroscopic (Infrared (IR), Electron Paramagnetic Resonance (EPR) techniques. The crystal structure of the complexes was identified by single crystal X-ray diffraction method.

14. Hui Liu [35] synthesized a Schiff base and three transition metal complexes of the base .The synthesized products were characterized by elemental analysis and spectroscopic techniques (IR, UV-Vis, NMR and mass).

15. Preparation, characterization and antimicrobial activity of a new Schiff base and its Vanadium (V) complex were investigated by X. W. Zhu [36]. The products have been characterized by several spectroscopic techniques including UV-Vis, IR and 1H NMR. By using single crystal X-ray diffraction method the crystal structure of the complex has also been identified.

16. Pakhontsu [37] synthesized N, N'-[4,4'-(Perfluoro- 1,4-phenylene) bis (oxy) bis (4,1-phenylene)] bis [2-(pyridin-2-ylmethylidene)-hydrazinecarbothioamide] . The author also prepared methyl and phenyl derivatives of the compound and their complexes with Cu and

Ni. The prepared complexes have biological functions and inhibit the development of the myeloid human leukemia HL-60 cancer cells.

17. Metal complexes of different metals with Schiff Bases have demonstrate the myriad of activities[38,39], and catalytic applications [40] in which metals arerepresented by Mo [41], Cu[50, 51], Zr [42–44], Pd [45], Cu [52], Ni [53], Ru [46], Mn [47],Zn [48] and W [49].

Objectives of the proposed study:

1. Synthesis of Schiff base metal complex:-

Preparation of mono/binuclear Schiff base metal complex by predictable methodology as mentioned in literature. Schiff base ligand is prepared by condensing aromatic amine, diketone (aliphatic / aromatic), aromatic aldehyde / ketone and other ligands. Copper(II), Nickel(II), Manganese(II), Cobalt(II), Zinc(II), Iron(II), Silver(II), Aluminium(III), and Chromium(III) salts/sulphates/acetate have been condensed with Schiff base ligands.

2. Application of analytical techniques:-

Analytical techniques such as elemental analysis, molar conductance, TLC and M.P

3. Spectral studies of metal complex:-

Characterization will be study by using infrared, ultra violet-visible, ^{1}H NMR, ^{13}C NMR, mass spectroscopy and thermal analysis. Furthermore, the crystals will be isolated by X-ray diffraction studies.

4. Study of some Pharmacological activity:-

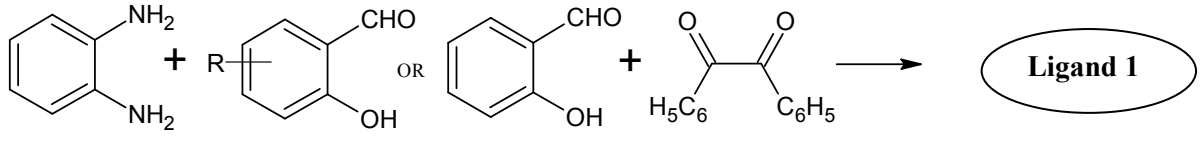
Antifungal, antibacterial etc.

Hypothesis/Assumptions:

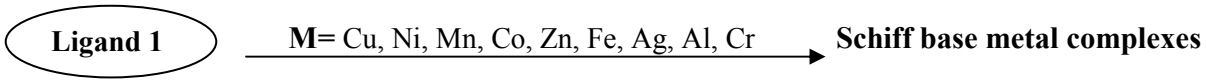
1. To find out new Schiff base and its metal complexes.
2. To find out more pharmacological applications.

Research methodology:

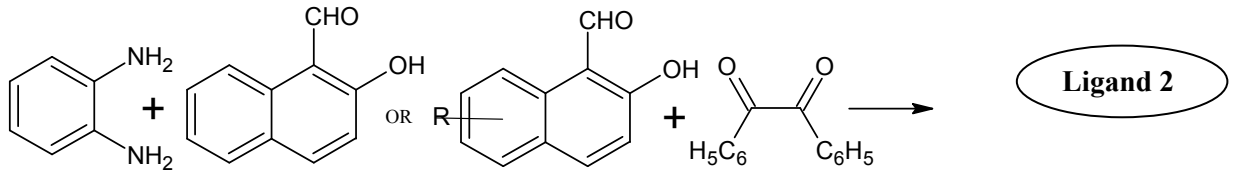
Scheme 1. Synthesis of ligand 1



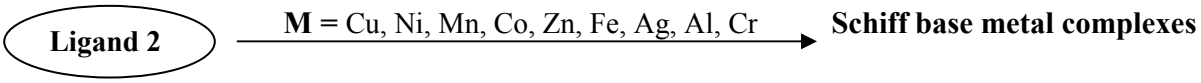
Scheme 1.1*Preparation of Schiff base metal complexes*



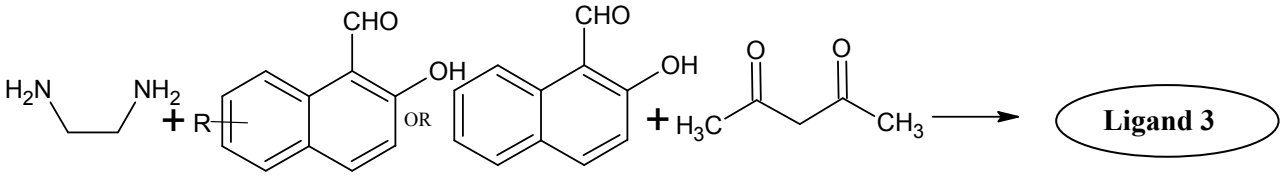
Scheme 2. Synthesis of ligand 2



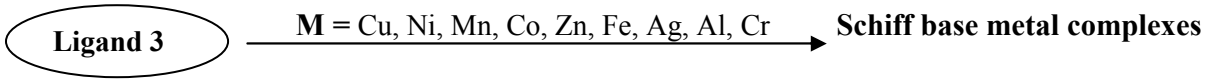
Scheme 2.1*Preparation of Schiff base metal complexes*



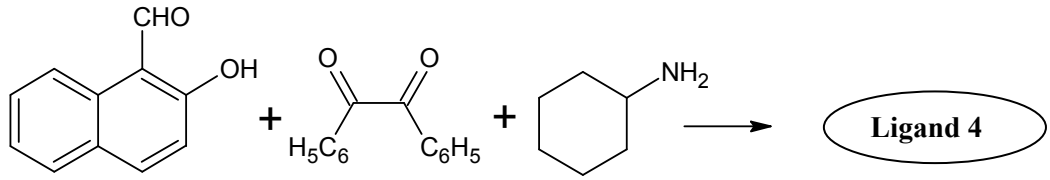
Scheme 3. Synthesis of ligand 3



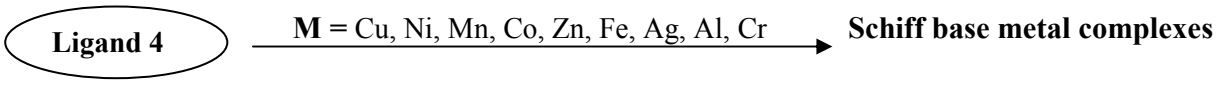
Scheme 3.1*Preparation of Schiff base metal complexes*



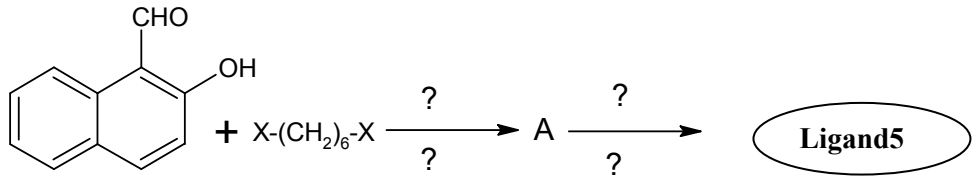
Scheme 4. Synthesis of ligand 4



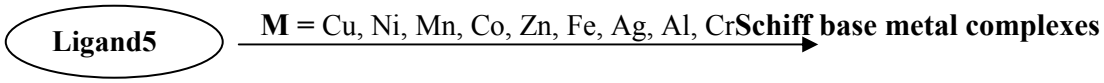
Scheme 4.1*Preparation of Schiff base metal complexes*



Scheme 5. Synthesis of ligand 5



Scheme 5.1*Preparation of Schiff base metal complexes*



Scope of Research Work:

The Schiff base ligands and their metal complexes have special importance in the field of co-ordination chemistry. A survey of the literature shows that, in recent times, increasing number of studies has been devoted to the synthesis and structural studies. However, the utility aspects of the Schiff bases and their metal complexes have received their share of attention with an all round progress in the field of co-ordination chemistry. The importance of Schiff bases and their metal complexes with transition metals have been well emphasized by several researchers.

The Schiff bases constitute one of the most active classes of the compounds possessing diversified biological applications such as antitubercular, anticancer, antibacterial, anti-inflammatory, antifungal, antitumor, diuretic, insecticidal, herbicidal, anthelmintic, anti-HIV, antiproliferative, anticonvulsant, antihypertensive and antiparasitic activities.

Chapter Scheme:

The tentative Chapter scheme:

Chapter - I Introduction and Literature Review

The introduction of the thesis will summarize this topic and its national and international status.

Chapter – II Scope and Objectives

Considering the use and special importance of the Schiff base ligands and their metal complexes in the field of co-ordination chemistry, an extensive study has been done with the increasing interest of these classical compounds behaving the diverse biological applications such as antibacterial, antifungal, antitubercular, anticancer, insecticidal etc.

Objectives of the study are given as follow:-

1) Preparation of mono/binuclear Schiff base metal complex by predictable methodology as mentioned in literature. Schiff base ligand is prepared by condensing aromatic / aliphatic amine, diketone (aliphatic / aromatic), aromatic aldehyde / ketone (Its derivatives) and other ligands. Copper(II), Nickel(II), Manganese(II), Cobalt(II), Zinc(II), Iron(II), Silver(II), Aluminium(III), and Chromium(III) salts/sulphates/acetate have been condensed with Schiff base ligands.

2) Analytical techniques such as elemental analysis, molar conductance, TLC and M.P

3) Characterization will be study by using infrared, ultra violet-visible, ^{1}H NMR, ^{13}C NMR, mass spectroscopy and thermal analysis. Furthermore, the crystals will be isolated by X-ray diffraction studies.

4) Pharmacological studies such as antifungal, antibacterial etc.

Chapter-III Experimental Techniques

The details of materials and methods used for work will be summarized in this topic. Synthetic starting material, reagents and solvents were of analytical reagent grade or of the highest quality commercially available and were dried when necessary.The progress of the reactions will be monitored by thin layer chromatography with silica-gel precoated sheets using proper solvent, UV light or iodine vapors are to be used for detection. IR spectra will be recorded, as KBr pellets, on FT-IR spectrophotometer with wave numbers given in cm^{-1}. Mass spectra obtain will be studied. ^{1}H NMR and ^{13}C NMR spectra along with chemical shift will be recorded and reported as δ relative to TMS as internal standard. Melting points (°C) were determined with an open glass capillary tube.

Chapter- IV Result and discussion

The results will be summarized in this topic and those will be discussed with appropriate references by giving suitable reasoning.

Chapter- V Summary and Conclusion

The summary and conclusion will be included in this topic with recommendations.

Chapter-VI References and Publications

The references cited in the introduction, materials & methods, and discussion will be summarized in this topic. The paper published in reputed journals.

Proposed work plan:

Months	Activities
1-6	Review of Literature, (Literature Survey), Study of various organic and inorganic reactions related to formation of Schiff base and its complexes with metals.
6-12	Designing of new methods for the synthesis of Schiff base metal complexes. Practical study of various catalysts, solvent and metals for the synthesis of different Schiff base metal complexes.
12-24	To develop newer efficient and to find its different new Pharmacological activities.
24-30	Data Compilation, tabulation, and analysis, seminar symposium, workshop.
30-36	Thesis writing and publication, interpretation, result compilation, participation.

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